Commonwealth of Massachusetts
Executive Office of Energy & Environmental Affairs

Department of Environmental Protection
One Winter Street Boston, MA 02108 • 617-292-5500

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MassDEP-Required Laboratory Procedures for Testing PFAS in Residuals

From: MassDEP Residuals Program and MassDEP Division of Environmental Laboratory Sciences, Senator William X. Wall Experiment Station

Date: February 8, 2021

Analysis of selected per- and polyfluoroalkyl substances (PFAS) in residuals (i.e., biosolids) from facilities that distribute/sell residuals in Massachusetts under an Approval of Suitability (AOS) can only be performed by a laboratory approved by MassDEP to perform this analysis.

General Information

In August 2020, MassDEP sent letters to all AOS holders requiring quarterly residuals testing for the following PFAS:

PFAS	Abbreviation	CASRN
Perfluorobutanoic acid	PFBA	375-22-4
Perfluoropentanoic acid	PFPeA	2706-90-3
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorononanoic acid	PFNA	375-95-1
Perfluorodecanoic acid	PFDA	335-76-2
Perfluoroundecanoic acid	PFUnA	2058-94-8
Perfluorododecanoic acid	PFDoA	307-55-1
Perfluorotridecanoic acid	PFTrDA	72629-94-8
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluoropentanesulfonic acid	PFPeS	2706-91-4
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3

Facilities must also submit all PFAS data generated according to the requirements of the U.S. EPA or other states.

Laboratory PFAS testing of residual samples shall include analysis of the solid product using appropriate analytical and quality control methods. PFAS testing results using the approved methodologies shall be submitted to MassDEP for quality assurance - quality control review and approval as soon as they are received from the laboratory. Please submit results to massdep.residuals@mass.gov both as a PDF of the laboratory report and as an Excel spreadsheet, unless otherwise directed by MassDEP.

Because there is no USEPA-approved method for testing residuals for PFAS, MassDEP has established a process to ensure that a laboratory will use appropriate analytical methods and quality control. MassDEP must review and approve the laboratory's proposed methodology, including detailed Standard Operating Procedures (SOPs) for PFAS testing of residual samples, and the Initial Demonstration of Capability (IDC) and Minimum Reporting Level (MRL) data demonstrating acceptable accuracy, precision, and MRLs in the analysis of PFAS-spiked blank Ottawa sand using the laboratory's SOP, before testing can begin.

A list of laboratories approved by MassDEP for testing residuals for PFAS is maintained here: https://www.mass.gov/info-details/testing-of-pfas-in-wastewater-and-residuals

In preparation for sampling, AOS holders may want to review the following PFAS sampling guidance documents:

> From the State of Michigan:
> https://www.michigan.gov/documents/pfasresponse/General_PFAS_Sampling_Guidance_634597_7.pdf
>
> From NEBRA:
> https://static1.squarespace.com/static/54806478e4b0dc44e1698e88/t/5ca2599a002291000145dd88/1554143647366/NEBRA-PFASSamplingAnalysisGuide_v.2-5Jan2018.pdf

If you have any questions regarding the MassDEP residuals program/AOS holders, please contact Jennifer Wood at Jennifer.wood@mass.gov.

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William X. Wall Experiment Station • 37 Shattuck Street, Lawrence MA 01843 • 978-682-5237

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Laboratory Requirements for Testing Residuals for PFAS

Residual sample collection and testing for PFAS must be performed according to the following guidelines:

1. For residual PFAS analysis, laboratories are expected to extract residual samples according to ASTM Method D7968 (modified by the laboratory as necessary) with subsequent extract PFAS analysis according to EPA Method 533 (modified by the laboratory as necessary). A laboratory must confirm that it meets a Minimum Reporting Level (MRL) for all target PFAS of less than or equal to 1.0 ng/g dry weight using spiked blank Ottawa sand (100% dry weight) as per the procedure in Section 9.1.4 of EPA Method 533. Also, a laboratory must minimize dilution of residual samples to only that which is necessary to handle significant matrix interference.

2. MassDEP expects that the facility will provide "Primary" and "Field Duplicate" residual samples (each with unique field and laboratory IDs) to the laboratory and that the laboratory will test both of these samples for PFAS using the laboratory's analytical method SOP approved by MassDEP for residuals PFAS analysis.

   The Chain-of-Custody (COC) Form submitted with the residual samples must be completed according to the example shown below. Note that the laboratory will consider the multiple containers with the same Client Sample ID as extra amount of the same identical sample.

Laboratory Sample ID	Client Sample ID	Client Sample Description	Sample Matrix	# of Containers	Container Volume	Container Type (Polypropylene)
XXXXX	Primary	Residual Type from Facility X	Solid	X		
XXXXX	Field Duplicate	Residual Type from Facility X	Solid	X		

Sample collection time must be different from the time the sample is relinquished to a shipping contractor or to the laboratory when directly delivering the samples to the laboratory.

3. MassDEP expects that with an extraction batch of 20 or fewer residual samples from one or more clients, the laboratory will select at least one of the residual samples as the Matrix Spike (MS) and MS Duplicate (MSD) or Laboratory Duplicate (LD) to be extracted, analyzed, and reported with the PFAS results for each of the residual samples in the extraction batch. MassDEP does not expect a facility to pay for these required QC samples even if the laboratory uses the facility's own residual sample for these QC analyses. AOS facilities must provide adequate sample amount for either the primary or field duplicate residual sample in case the MS and MSD or LD in an extraction batch is tested using that sample from an AOS holder. With the understanding that the amount of sample necessary varies depending on the aqueous nature of the residual, the amount of additional sample provided to the laboratory may vary and may include more than one container of the primary and/or field duplicate sample(s); note that the additional sample containers must be labeled with the same field and laboratory IDs as the corresponding primary or field duplicate sample.

4. An AOS holder may choose but is not required to submit a field reagent blank (FRB) with the primary and field duplicate residual samples to meet MassDEP's requirements for testing PFAS in residuals. An FRB is a reagent water sample that is transferred from one bottle to a clean bottle at a PFAS sampling site and is therefore exposed to the field environment – it is used to determine if PFAS samples could have been contaminated in the field during the sampling process. Note that a trip blank (i.e., a reagent water sample in a sealed bottle that travels to the field and then back to the laboratory unopened with the field samples) is not necessary for PFAS analysis.

5. MassDEP expects that laboratory reports detailing PFAS in residuals will include analytical data for all applicable MS and MSD or LD performed in the same extraction batch(es) (as described above) as the residual samples. Note that MassDEP (currently in consultation with Tetra Tech) is reviewing each laboratory report of residual PFAS analysis. Providing acceptable QA/QC data will be critical to the approval of residuals PFAS analytical data.

If you have any laboratory-related questions on testing residuals for PFAS, please contact Lisa Touet at 978-242-1364 or lisa.touet@mass.gov.